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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : <b>C11D</b>	<b>A2</b>	(11) International Publication Number: <b>WO 00/02980</b> (43) International Publication Date: 20 January 2000 (20.01.00)
<p>(21) International Application Number: PCT/GB99/02048</p> <p>(22) International Filing Date: 8 July 1999 (08.07.99)</p> <p>(30) Priority Data: 9814968.5 10 July 1998 (10.07.98) GB</p> <p>(71)(72) Applicant and Inventor: HINTON, Gerald, Thomas [GB/GB]; Eastfields, Stonebyres, Lanark ML11 9UW (GB).</p> <p>(74) Agent: MURGITROYD &amp; COMPANY; 373 Scotland Street, Glasgow G5 8QA (GB).</p>		<p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BI, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>Without international search report and to be republished upon receipt of that report.</i></p>
<p>(54) Title: DETERGENT</p> <p>(57) Abstract</p> <p>A detergent composition comprising a granulated percarbonate and a blend which encapsulates the percarbonate is described. The blend comprises a sulphate, carboxymethyl cellulose and a nonionic surfactant. The detergent composition comprises sodium metasilicate and does not include a zeolite, a perborate or a phosphate. The composition is capable of being stored in a water-soluble PVA film packaging for at least nine months and wherein the composition comprises between 1 % and 15 % percarbonate. The composition can include a phosphate substitute such as a polyacrylate. The composition can be compressed into a tablet format and used as a laundering product.</p>		

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1     "Detergent"

2

3     This invention relates to a detergent product  
4     formulations which can be packaged in water soluble  
5     film. The invention also relates to detergent  
6     formulations excluding phosphates.

7

8     A product of the invention is ideally for use in the  
9     laundering and conditioning of industrial and domestic  
10    man-made and/or natural fabrics in semi-automatic or  
11    automatic washing machines. It may also be used in  
12    dishwashers. For convenience purposes it is useful if  
13    this can be achieved by means of a soluble single  
14    compartment sachet containing varying amounts of  
15    bleaching detergents including for example sodium  
16    percarbonate. Typically a sachet may be made from a  
17    water soluble film such as PVA.

18

19    Conventional laundering detergents comprise perborates  
20    and zeolites and these compounds are not stable in  
21    water soluble film. Previous attempts have been made  
22    to manufacture fully built detergent and conditioner  
23    systems in a sachet have required a twin compartment  
24    sachet manufactured from a perforated film. These  
25    fully built detergent and conditioner systems generally

1 contain bleaching agents. Sodium percarbonate is  
2 recognised in this field as a bleaching agent.  
3 However, use of percarbonate in sachets is not popular  
4 as it is unstable when combined with other components  
5 of a high moisture content.

6  
7 Twin compartment sachets have a disadvantage in that  
8 they require greater mechanical action to dissolve the  
9 sachet and thus have long dispersion times. Also, they  
10 are expensive to manufacture.

11  
12 Additionally, the perforated film used in these twin  
13 compartment sachets does not confer a significant shelf  
14 life to the components contained within the sachet,  
15 wherein the oxidising power of the bleaching agent is  
16 reduced.

17  
18 Also, typically these formulations contain zeolites.  
19 These have high moisture content which affects the  
20 mechanical properties of the film e.g. the pliability.

21  
22 Pollution problems arise from the use of phosphates in  
23 detergent compositions. Phosphates are required as  
24 solubilisers and aid detergency. However, they have a  
25 detrimental effect on the environment. Forthcoming  
26 European legislation will require the amount of  
27 phosphate released into the environment to be  
28 minimised.

29  
30 It is an object of the present invention to provide a  
31 detergent or bleaching agent for cleaning in laundries,  
32 or in domestic washing machines or dishwashers which is  
33 phosphate free.

34  
35 According to one aspect of the present invention there  
36 is provided a detergent composition comprising a

1 granulated percarbonate and a blend which encapsulates  
2 the percarbonate, the blend comprising a sulphate,  
3 carboxymethyl cellulose and a nonionic surfactant,  
4 wherein the detergent composition comprises sodium  
5 metasilicate and does not include a zeolite, a  
6 perborate or a phosphate, and wherein the composition  
7 is capable of being stored in a water-soluble PVA film  
8 packaging for at least nine months and wherein the  
9 composition comprises between 1% and 15% percarbonate.

10

11 Preferably the detergent formulation uses sodium  
12 percarbonate, carboxy methyl cellulose, sodium  
13 sulphate, a nonionic surfactant blend, sodium silicate  
14 and a phosphate substitute, ie an alternative to the  
15 phosphates used in detergent formulations.

16

17 The phosphate substitute may be selected from the group  
18 comprising silicates, carbonates and polycarboxylates,  
19 which should be wholly or substantially anhydrous.

20

21 The invention specifically excludes polyphosphates and  
22 preferably includes polyacrylates in powdered or liquid  
23 form. In particular formulations the amount of  
24 emulsifier metasilicate is also increased and the  
25 amount of carboxymethyl cellulose is decreased and  
26 bleach activator is added when liquid polyacrylate is  
27 used.

28

29 Preferably the detergent further comprises bleach  
30 activator such as TAED.

31

32 The detergent may further comprise at least one  
33 ingredient chosen from the group comprising linear  
34 alkylbenzene sulphonate, sodium lauryl sulphate, sodium  
35 carbonate, low foam wetting agent, perfumes, optical  
36 brighteners, salts, pigments and enzymes.

1 In one embodiment the detergent formulation is a  
2 laundering product.

3  
4 In an alternative embodiment the detergent is a machine  
5 dishwashing product.

6  
7 Suitably the laundry or dishwashing product according  
8 to the present invention is packaged in PVA film. A  
9 product of the invention is stable in PVA film compared  
10 to other products containing zeolites and perborates.

11  
12 Suitably the film is 20-80 microns thick.

13  
14 The product may be incorporated into a tablet form.

15  
16 The granulated form of percarbonate in the above  
17 permits efficient bleaching action of the laundry  
18 product whilst not effecting the stability of the  
19 product in storage.

20  
21 While modifications and improvements may be made  
22 without departing from the scope of this invention, the  
23 following is a description of the invention, with  
24 reference to the accompanying diagrams:

25  
26 Figures 1a and 1b illustrate a soluble single  
27 compartment sachet produced from a polyvinyl alcohol  
28 (PVA) film filled with product and heat sealed.

29  
30 Figures 2a and 2b illustrate a soluble single  
31 compartment sachet produced from PVA film by  
32 thermoforming.

33  
34 The sachets are sealed such that they contain a laundry  
35 and conditioning powder without spillage or air borne  
36 contamination which can cause irritation to eyes and/or

1 skin etc.

2

3 Example 1

4

5 The laundry and conditioning powder can be in the form  
6 of a super concentrate with a bulk density of not less  
7 than 0.75kg/l. The laundry and conditioning powder is  
8 preweighed and packed in 50g batches which is  
9 sufficient to launder 4.5kg dry weight of mixed fibres  
10 (normal soiling) in either hard or soft water  
11 conditions.

12

13 In order to determine the storage and durability of  
14 sachets containing laundry and conditioner, the sachets  
15 were treated as follows:

16

17 1. Laundry and conditioner products including the  
18 granulated percarbonate compound were sealed in  
19 PVA sachets under atmospheric conditions and  
20 stored in various temperatures.

21

22 2. Sachets containing the laundry and conditioner  
23 products were sealed in a PVC container under  
24 atmospheric conditions as stored at various  
25 temperatures.

26

27 The samples of both 1 and 2 above were stored for nine  
28 months whereupon they were added to separate washing  
29 cycles. In both cases the samples were found to be  
30 stable (both before use and after storage) with no  
31 deterioration of the product or the sachet containing  
32 the product.

33

34 Sachets were dissolved in cold water (20°C) using a  
35 combination of water flow and mechanical agitation  
36 whereupon sachets and contents were typically

1 completely dissolved with no residue within 90 seconds.

2

3 The polyvinyl alcohol film was 30-85 microns (+/- 10-  
4 15%) thick. The polyvinyl alcohol film is both  
5 biodegradable and nonhazardous.

6

7 The process for producing the sachets according to  
8 figures 1a and b containing the dishwashing, laundry  
9 and/or conditioner product requires a form filling  
10 machine modified such that the sachet is produced with  
11 a minimum number of folds and seals.

12

13 Alternatively thermoforming of film can be used to  
14 produce filed sachets as illustrated in Figure 2.

15



## 1     Example 2

2

## 3     Typical detergent product formulations

4

5	Linear alkylbenzene sulphonate (LABS)	0.5%
6	Sodium Percarbonate	1-15%
7	Carboxy Methyl Cellulose (CMC)	1-5%
8	Sodium Sulphate Anhydrous	5-35%
9	Sodium Carbonate	0-35%
10	Nonionic Surfactant Blend	1-10%
11	Low Foam Wetting Agent	0-2%
12	Sodium Metasilicate	1-30%
13	Perfumes	0-1.5%
14	Optical Brighteners	0-1%
15	Salts	0-10%
16	Enzymes (blended)	0-5%
17	Copolymer	0-10%
18	Water Soluble Dye Pigment	0-2%
19	Sodium Polyacrylate	1-30%
20	(eg Sandoperol)	(preferably
21		2-12%)
22	Bleach Activator	0.1-2%

23

24     Minor ingredients as required.

25

26     Linear alkylbenzene sulphonate may be replaced with 0.5  
27     to 10% sodium lauryl sulphate to produce a more  
28     environmentally friendly product.

29

30     Varying amounts of the above components may be used  
31     depending on the type of product required, i.e. for  
32     laundering, dishwashing or conditioning.

33

34     In the following examples nonoinic surfactant blend and  
35     low foam wetting agent are together referred to as  
36     liquid blend.

## 1     Example 3

2

## 3     Laundry Product using Liquid Polyacrylate

4

5     A laundering product was prepared and packaged in PVA  
6     film.

7

8     The formulation consisted of

9

10	Linear alkylbenzene sulphate	2%
11	Sodium Percarbonate	3%
12	Carboxy Methyl Cellulose	1%
13	Sodium Sulphate Anhydrous	25%
14	Sodium Carbonate	28%
15	Liquid blend	2%
16	Sodium Metasilicate	30%
17	Anionic Sodium Polyacrylate	10%
18	Perfumes	0.8%
19	Optical Brighteners	0.5%
20	Salts	2%
21	Enzymes (blended)	1.5%
22	Copolymer	2%
23	Bleach Activator (TAED)	1%

24

25     Prior to mixing the liquid polyacrylate is blended with  
26     approximately 60% of the anhydrous sulphate and dried.

27

28     Alternatively, the polycrylate may be mixed with any of  
29     the powdered materials, dispersed through the powder.

30

## 31     Example 4

32

## 33     Laundry Product

34

35     A laundering product was prepared and packaged in PVA  
36     film.

1 The formulation consisted of

2		
3	Linear alkylbenzene sulphate	2%
4	Sodium Percarbonate	5.1%
5	Carboxy Methyl Cellulose	1.0%
6	Sodium Sulphate Anhydrous	20%
7	Sodium Carbonate	33.0%
8	Liquid blend	2.0%
9	Sodium Metasilicate	30%
10	Perfumes	0.8%
11	Optical Brighteners	0.5%
12	Salts (NTA Powder)	2%
13	Enzymes (blended)	1.5%
14	Copolymer	2.0%
15	Bleach Activator (TAED)	1%

16

17 Inclusion of copolymer improved redeposition.

18

19 Production of Formulation

20

21 The percarbonate was added to the sachet as shown in  
22 Figure 1 in the form of granules. These granules  
23 comprised percarbonate, sulphate and carboxy methyl  
24 cellulose in varying amounts together with a blend of  
25 nonionic surfactants to create a binding agent. These  
26 components were processed in order to produce a dust  
27 free granule of a diameter not less than 150 microns.

28

29 In order to produce the granules a horizontal type  
30 mixer was used. A liquid blend of the abovementioned  
31 laundry components was added to the mixer from a high  
32 pressure vessel incorporating an agitator. The liquid  
33 blend was fed in at a pressure of 60 pounds per square  
34 inch.

35

36

1 The finished granulated detergent is fully  
2 biodegradable and has a stable pH range of 10-11, which  
3 does not affect the PVA film stability as used in this  
4 invention.

5  
6 Trials have shown that using nonionic surfactants  
7 comprising alkyl aryl polyglycol ethoxylates through  
8 the alkyl group C<sub>6-12</sub> (typically C<sub>8-10</sub>) is stable and  
9 gives the best results even after storage in excess of  
10 9 months.

11  
12 A typical encapsulation blend is as follows:

13  
14 Sodium Sulphate (Anhydrous) 5-98%  
15 Carboxy Methyl Cellulose 1-25%  
16 Nonionic Surfactant blends 1-40%

17  
18 Alternative nonionic surfactant blends comprising  
19 alcohol polyglycol ethoxylate oxide in the range of  
20 0.5-5% have been used successfully.

21  
22 The advantages of the invention and of the ways in  
23 which the disadvantages of the previously known  
24 arrangements are overcome include encapsulation of a  
25 percarbonate with a powder/liquid blend forming a  
26 granular product of suitable size and strength for use  
27 in a hot or cold process.

28  
29 A single component sachet sealed such that the  
30 percarbonate does not decompose in the detergent  
31 contained within the sachet.

32  
33 Upon dissolution the PVA leaves no residues i.e. it is  
34 fully dissolved.

35  
36

1 No mechanical action is required to dissolve the PVA  
2 film.

3  
4 The encapsulation process extends the shelf life of  
5 fully built detergent within the PVA sachet.

6  
7 In the super concentrated form, a laundering  
8 formulation normally requires 50g per 4.5kg (dry  
9 weight) wash with normal soiling.

10

11 Example 5(a)

12

13	Sodium Sulphate	20	%
14	Sodium Carbonate	40	%
15	Sodium Metasilicate Anhydrous	15	%
16	Sodium Percarbonate Peroxyhydrate	10	%
17	CMC	1	%
18	Alcohol Polyglycol Ethoxylate		
19	Oxide + Cationic Resin Dye		
20	Transfer Inhibitor	5	%
21	LABS	1.2	%
22	NTA Powder/EDTA Powder	5	%
23	Enzymes (optional)	0.5	%
24	Co Polymer	1	%
25	TAED	1	%
26	OBA (optional)	0.3	%
27	Perfume (optional)	-	

28

29 Example 5(b)

30

31	Sodium Sulphate	14	%
32	Sodium Carbonate Dense	50	%
33	Sodium Metasilicate Anhydrous	10	%
34	Alcohol Polyglycol ether +		
35	Cationic resin Dye		
36	Transfer Inhibitor	10	%

1	CMC	2 %
2	Percarbonate	4.5%
3	Optical Brightners	0.3%
4	LABS	1.2%
5	NTA Powder (salts)	5 %
6	Perfumes	0.5%
7	Enzymes	0.5%
8	Co Polymer	1 %
9	TAED	1 %

10

11 In the above Examples, no phosphate or phosphate  
12 substitute is used. The level of sodium carbonate is  
13 increased in Example 5(b) to act as a filler and  
14 provides the alkalinity previously provided by  
15 phosphate. An increased level of percarbonate acts  
16 similarly.

17

## 18 Example 6

19

20	Sodium Metasilicate	18 %
21	Sodium Carbonate	40 %
22	Sodium Sulphate	14 %
23	Sodium Polyacrylate Acid	6 %
24	Sodium Percarbonate	8 %
25	CMC	1 %
26	NTA/EDTA Powder	4 %
27	LABS	2 %
28	Perfume	0.8%
29	Liquid Alcohol Polyglycol Ether +	
30	Dye Transfer Inhibitor	3 %
31	Enzymes	0.6%
32	OBAs	0.3%
33	Co Polymer	1 %
34	TAED	1.3%

35

36

1     **CLAIMS**

2

- 3     1.    A detergent composition comprising a granulated  
4            percarbonate and a blend which encapsulates the  
5            percarbonate, the blend comprising a sulphate,  
6            carboxymethyl cellulose and a nonionic surfactant,  
7            wherein the detergent composition comprises sodium  
8            metasilicate and does not include a zeolite, a  
9            perborate or a phosphate, and wherein the  
10           composition is capable of being stored in a water-  
11           soluble PVA film packaging for at least nine  
12           months and wherein the composition comprises  
13           between 1% and 15% percarbonate.
- 14
- 15     2.    A composition as claimed in claim 1 wherein the  
16           percarbonate is sodium percarbonate.
- 17
- 18     3.    A composition as claimed in claim 1 or claim 2  
19           wherein the sulphate is sodium sulphate.
- 20
- 21     4.    A composition as claimed in any of the preceding  
22           claims wherein the surfactant is alkyl (C<sub>6</sub> to C<sub>12</sub>)  
23           aryl polyglycol ethoxylate.
- 24
- 25     5.    A composition as claimed in any of the preceding  
26           claims wherein the composition further comprises  
27           at least one of the ingredients chosen from the  
28           group comprising linear alkylbenzene sulphonate,  
29           sodium carbonate, low foam wetting agent,  
30           perfumes, cationic surfactant, optical  
31           brighteners, salts and enzymes.
- 32
- 33     6.    A composition as claimed in any one of the  
34           preceding Claims wherein the composition further  
35           includes a phosphate substitute.
- 36

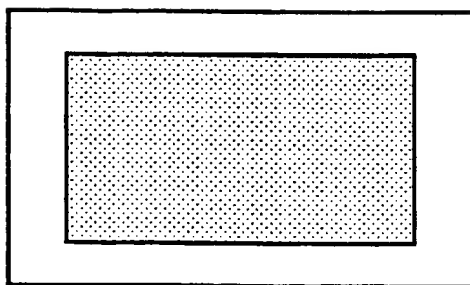
- 1     7.    A composition as claimed in Claim 6 wherein the  
2           phosphate substitute is selected from the group  
3           comprising silicates, carbonates and  
4           polycarboxylates.  
5
- 6     8.    A composition as claimed in Claim 7 wherein the  
7           phosphate substitute is a polyacrylate.  
8
- 9     9.    A composition as claimed in Claim 8 wherein the  
10          phosphate substitute is sodium polyacrylate.  
11
- 12    10.   A composition as claimed in any of the preceding  
13          claims wherein the composition is a laundering  
14          product.  
15
- 16    11.   A composition as claimed in any of the preceding  
17          claims wherein the composition is a machine  
18          dishwashing product.  
19
- 20    12.   A composition as claimed in any one of the  
21          preceding claims wherein the PVA film is 20-80  
22          microns thick.  
23
- 24    13.   A composition as claimed in any one of the  
25          preceding claims wherein the product is compressed  
26          into a tablet format.  
27



$2/2$

Figure 2

(a)



(b)

